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SPECIFICATION FOR OLEIC ACID, TECHNICAL

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *November* 1960

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Indian Standard
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AMENDMENT NO. 2 FEBRUARY 1976
TO
IS : 1676-1960 SPECIFICATION FOR OLEIC ACID, TECHNICAL

Through this amendment a requirement and method of test for fluxing property of the material is being included in this standard on a request from the Ministry of Defence, Government of India. The amended standard, therefore, achieves full alignment with JSS : 1076 Oleic acid, commercial (flux soldering) of Ministry of Defence.

Alteration

[*Page* 4 (*page* 5 *of the reprint*), *Table* I, *clause reference under the title of the table*] — Substitute ' (*Clauses* 4.3 *and* 5.1) ' *for* ' (*Clauses* 4.2 *and* 5.1) '.

Addenda

[*Page* 3 (*page* 4 *of the reprint*), *clause* **4.1.1**] — Add the following new clause after **4.1.1** and re-number the existing clause '**4.2**' as '**4.3**':

'**4.2 Fluxing Property** — The material, when intended for Defence use, shall also pass the test prescribed in Appendix C.'

(*Page* 12, *clause* **B-3.2.3**) — Add the following new appendix after **B-3.2.3**:

APPENDIX C

(*Clause* 4.2)

TEST FOR FLUXING PROPERTY

C-1. OUTLINE OF THE METHOD

C-1.1 Material is applied to two properly cleaned test plates by brush and plates are put one over the other and soldered with soldering materials. The plates are then examined for any detachment.

C-2. MATERIALS

C-2.1 Tin Plates — two of 10 × 2·5 cm size.

C-2.2 Solvents — suitable, like acetone, carbon tetrachloride or toluene.

C-2.3 Solder Gun

C-2.4 Soldering Materials — suitable.

C-3. PROCEDURE

C-3.1 Take the tin plates and polish them with emery paper. De-grease them with a suitable solvent and finally clean with dry cloth.

C-3.2 Apply oleic acid by brush on the part of the surface of the plates to be soldered. Put the soldering material on the portion to be soldered and spray it by red hot solder gun. Put the plates one over the other and allow the soldered plates to cool for 2 to 3 minutes. The soldered portion shall not get detached under normal stress.

C-3.3 The test shall be taken as satisfactory if the plates do not get separated easily when tried to break off the soldered joint.

(CAFDC 5)

Reprography Unit, BIS, New Delhi, India

Indian Standard
SPECIFICATION FOR OLEIC ACID, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 4 July 1960, after the draft finalized by the Oils, Fats and Soaps Sectional Committee had been approved by the Chemical Division Council and the Agricultural and Food Products Division Council.

0.2 Oleic acid is used in India in the manufacture of various products, such as lubricants, carbon paper, stencil paper, paints, cosmetics, liquid soaps, disinfectants, textile finishing preparations, printing inks, pharmaceuticals, metal polishes, etc. Although there are many commercial specifications drawn up by individual manufacturers which relate to different grades of oleic acid, these have no official recognition and no standard has yet been formulated for this material by any country. In preparing this standard, due consideration has been given to the different grades of oleic acid being used at present for various purposes.

0.3 Taking into consideration the views of producers, consumers and technologists, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the manufacturing, trade and technological procedures followed in the country in this field.

0.4 In the preparation of this standard, substantial assistance has been derived from the valuable data made available by various organizations in this country, notably Calcutta Chemical Co Ltd, Calcutta; Godrej Soaps Private Ltd, Bombay; Hindustan Lever Ltd, Bombay; Modi Vanaspati Manufacturing Co Ltd, Modinagar; Navsari Oil Products Ltd, Navsari; and Standard-Vacuum Oil Company Ltd, Bombay. The following publications were also referred to:

British Pharmacopoeia, 1958
United States Pharmacopoeia, 1955

0.5 Indian Standard Methods of Sampling and Test for Vegetable Oils and Fats (IS : 548-1954*) is a necessary adjunct to this standard. Besides, this standard requires reference to IS : 1070-1960 Specification for Water, Distilled Quality (*Revised*).

0.5.1 Wherever a reference to any Indian Standard mentioned in **0.5** or otherwise appears in this standard, it shall be taken as a reference to the latest version of the standard.

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*Since revised.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960 Rules for Rounding Off Numerical Values (*Revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

0.7 This standard is intended chiefly to cover the technical provisions relating to the material, and it does not include all the necessary provisions of a contract.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for oleic acid, technical.

2. GRADES

2.1 The material shall be of three grades, namely, Grade 1, Grade 2 and Grade 3.

2.1.1 Grade 1 is recommended for use in the manufacture of cosmetics.

3. SAMPLING

3.1 Representative samples of the material shall be drawn as prescribed in Appendix A.

4. REQUIREMENTS

4.1 Description — Oleic acid, technical, shall be the product obtained by the hydrolysis of fats or fixed oils and the subsequent separation of the liquid fatty acids. It shall be clear and free from adulterants, sediment, suspended and other foreign matter.

4.1.1 The clarity of the material shall be judged by the absence of turbidity after keeping the sample at 27° ± 2°C for 24 hours.

4.2 The material shall also comply with the requirements given in Table I.

5. TESTS

5.1 Tests shall be carried out by methods prescribed in col 6 and 7 of Table I.

6. PACKING AND MARKING

6.1 The material shall be supplied in suitable containers, as agreed between the purchaser and the supplier.

TABLE 1 REQUIREMENTS FOR OLEIC ACID, TECHNICAL

(*Clauses* 4.2 *and* 5.1)

Sl No.	Characteristic	Requirement for			Method of Test, Ref to Cl No. in	
		Grade 1	Grade 2	Grade 3	IS : 548-1954†	Appendix B
1)	(2)	(2)	(4)	(5)	(8)	(7)
i)	Moisture, percent by weight, *Max*	0·1*	0·25	0·25	4	—
ii)	Saponification value, *Min*	200	197	194	14	—
iii)	Acid value shall not differ from saponification value by more than	4	6	8	6	—
iv)	Iodine value	90 to 95	85 to 95	85 to 95	13	—
v)	Mineral acidity	Nil	Nil	Nil	—	B-1
vi)	Ash, percent by weight, *Max*	0·1	0·1	0·25	—	B-2
vii)	Unsaponifiable matter, percent by weight, *Max*	1·0	1·5	1·5	7	—
viii)	Pour point (°C), *Max*	15	20	27	—	B-3
ix)	Colour reading on the Lovibond scale, *Max*	15·0 Y + 1·5 R in 5¼-in cell	10·0 Y + 2·0 R in 1-in cell	—	12	—

*For cosmetic use no moisture limit is prescribed.

†Since revised.

6.2 The containers shall be securely closed and legibly and indelibly marked with the following information:

a) Manufacturer's name;
b) Recognized trade-mark, if any;
c) Name and grade of the material;
d) Weight of the material in the container;
e) Month and year of the manufacture; and
f) Batch number in code or otherwise to enable the lot of manufacture to be traced back from records.

6.2.1 The containers may also be marked with Standard mark.

6.2.2 The use of the Standard Mark is govered by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

A-3.4 Referee Sample — The third set of the test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee sample and shall be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier.

A-4. NUMBER OF TESTS

A-4.1 The test for saponification value, acid value and pour point (*see* Table I) shall be carried out on each individual sample of the set of test samples (*see* **A-3.3**).

A-4.2 The tests for the remaining characteristics given in Table I shall be carried out on the composite sample of the set of test samples (*see* **A-3.3**).

A-5. CRITERION FOR CONFORMITY

A-5.1 A lot shall be considered as conforming to this specification if it satisfies the criteria in **A-5.2.1** and **A-5.3** for the characteristics given in Table I.

A-5.2 The test results for saponification value, acid value and pour point shall be recorded as shown in Table III. The mean and the range shall be calculated as follows and shall be recorded in col 4 and 5 respectively of Table III:

$$\text{Mean } (\bar{x}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}$$

Range (R) = The difference between the maximum and the minimum values of the test results

A-5.2.1 The corrected mean as shown in col 6 of Table III shall be calculated. The lot shall be considered to have satisfied the requirement for a characteristic if the condition given in col 7 of Table III is satisfied.

TABLE III CRITERION FOR CONFORMITY

(*Clauses* A-5.2 *and* A-5.2.1)

Sl No.	Characteristic	Test Results 1, 2, 3...	Mean	Range	Corrected Mean	Criterion for Conformity
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Saponification value		$\bar{x}_1$	R_1	$\bar{x}_1 - 0{\cdot}6\ R_1$	Corrected mean ≥ specified value in Table I (ii)
ii)	Acid value		$\bar{x}_2$	R_2	$\bar{x}_2 - 0{\cdot}6\ R_2$	Corrected mean ≥ specified value in Table I (iii)
iii)	Pour point		$\bar{x}_3$	R_3	$\bar{x}_3 + 0{\cdot}6\ R_3$	Corrected mean ≤ specified value in Table I (viii)

A-5.3 The composite sample when tested for the remaining characteristics not tested in **A-5.2** shall satisfy the corresponding requirements for them as specified in Table I.

APPENDIX B

(*Table* I)

TESTS FOR MINERAL ACIDITY, ASH AND POUR POINT

B-1. TEST FOR MINERAL ACIDITY

B-1.1 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

B-1.2 Reagents — The following reagents are required.

B-1.2.1 *Methyl Orange Indicator* — 0·05 percent (*w/v*) solution.

B-1.2.2 *Light Petroleum Ether* (60°/80°C).

B-1.3 Procedure — Measure 10 ml of the melted sample into a separating funnel and shake intimately with three successive 10 ml portions of hot water. The temperature of the hot water should be more than the melting point of oleic acid. Combine the aqueous extracts, transfer to another separating funnel and remove traces of fatty acids in the water by extraction with light petroleum ether. Test the aqueous extract so obtained with a few drops of methyl orange indicator.

B-1.4 The material shall be taken to have satisfied the requirements of the test if the indicator does not show acid reaction.

B-2. DETERMINATION OF ASH

B-2.1 Apparatus

B-2.1.1 *Platinum Crucible*

B-2.1.2 *Desiccator* — containing an efficient desiccant, such as fused calcium chloride.

B-2.2 Procedure — Weigh accurately about 10 g of the air-dried material into a platinum crucible which has been previously dried, cooled in the desiccator and weighed. Heat the crucible over a low flame and ignite the contents gently. Incinerate the residue in a muffle furnace at 550° ± 10°C until free from carbon. Cool the crucible in a desiccator and weigh. Repeat the above procedure of heating, cooling and weighing until the difference between two successive weighings does not exceed 1 mg.

B-2.3 Calculation

$$\text{Ash, percent by weight} = \frac{100\,w}{W}$$

where

w = weight in g of the ash, and

W = weight in g of the material taken for the test.

B-3. DETERMINATION OF POUR POINT*

B-3.1 Apparatus — The apparatus consisting of the following parts assembled as shown in Fig. 1 shall be used.

B-3.1.1 *Jar A* — of clear glass, cylindrical form with a flat bottom, 115 to 125 mm high and 30 to 33·5 mm inside diameter fitted with a cork *C* bored centrally to carry the thermometer *B*.

B-3.1.2 *Thermometer B*† — conforming to the following requirements:

Range	−38°C to +50°C
Graduation	1°C
Immersion	108 mm
Overall length	225 ± 10 mm
Stem diameter	5·5 to 8·0 mm
Bulb shape	Cylindrical
Bulb length	7 to 10 mm
Bulb diameter	Not greater than stem
Length of graduated portion	65 to 85 mm
Distance between bottom of bulb to − 38°C mark, *Min*	120 mm
Longer lines at each	5°C from −35°C
Figured at each	10°C from −30°C
Expansion chamber	To allow heating to 100°C
Top finish	Plain
Scale error not to exceed	±0·5°C

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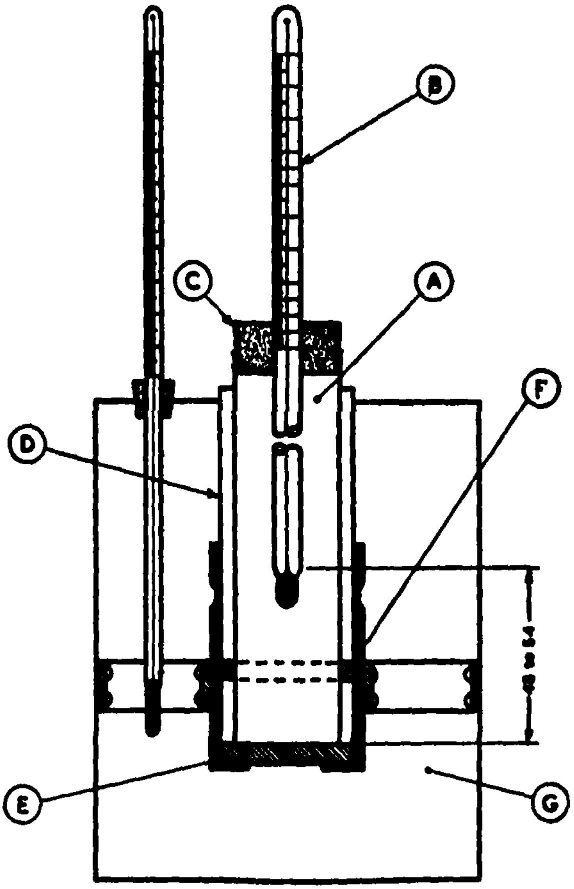
*The method is based on P : 10 Cloud Point and Pour Point given in IS : 1448 (Part I)-1960 with suitable modification.

†Mercury-in-glass type thermometer with Institute of Petroleum designation IP 1C conforms to these requirements.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF POUR POINT

B-3.1.3 *Jacket D* — of glass or metal, water-tight, cylindrical in form, flat-bottom, about 115 mm in depth with inside diameter 9·5 to 12·5 mm greater than the outside diameter of the jar *A*. A disc of cork or felt *E*, 6 mm thick, fits snugly in the bottom of the jacket.

B-3.1.4 *Gasket F* — in the shape of a ring about 5 mm thick fitting snugly round the jar *A* and loosely inside the jacket *D*. It may be made of cork, felt, or any other suitable material elastic enough to cling to the jar *A* and hard enough to hold its shape. The gasket prevents the jar from touching the jacket.

B-3.1.5 *Bath G* — of any size and shape convenient for obtaining the required temperature, and equipped with a support which will hold jacket *D* firmly in a vertical position.

B-3.1.5.1 It is convenient to have two or more baths available when determining pour points below 10°C.

B-3.1.5.2 The bath liquid may be maintained at the required temperature by refrigeration, if available, taking care to avoid disturbance of the bath liquid and also avoiding direct contact of any immersed cooling coils with the jacket. Alternatively, one of the following freezing mixtures may be used in the bath:

Freezing Mixture	*For Temperature Down to*
Crushed ice and water	10°C
Crushed ice and sodium chloride	—12°C
Crushed ice and calcium chloride crystals	—25°C
Solid carbon dioxide and acetone	—55°C

B-3.2 Procedure

B-3.2.1 Pour the material, heated in a water-bath, if necessary, into the jar to a height of 51 to 57 mm (*see* **B-3.2.1.1**). Close the jar with the cork carrying the thermometer so that the thermometer bulb is immersed vertically in the material with the beginning of the capillary 3 mm below the surface. Heat the material, without stirring, to a temperature of 46°C in a bath maintained at a temperature not higher than 48°C. Cool the sample to 32°C in air or in a water-bath at approximately 25°C. Fit the gasket on to the jar 25 mm from the bottom and insert the jar into the jacket. The disc, the gasket and the jacket shall be kept clean and dry.

B-3.2.1.1 The jar may be marked to indicate the proper level of the sample.

B-3.2.2 Once the material has cooled enough to allow the formation of wax crystals, take great care not to disturb the mass of sample nor to permit the thermometer to shift in the sample. Any disturbance of the spongy network of crystals will lead to false results.

B-3.2.3 Maintain the bath temperature of the cooling bath at minus 1° to plus 12°C. Support the jacket and jar in a vertical position in the bath so that not more than 25 mm projects from the cooling medium. Beginning at a temperature 12°C before the expected pour point, at each thermometer reading which is a multiple of 3°C, remove the jar from the jacket carefully and tilt it just enough to ascertain whether there is a movement of the material, and replace it; this complete operation, removal and replacement should take not more than three seconds. Take care not to disturb the mass of the liquid nor to permit the thermometer to shift in the liquid. If the material has not ceased to flow when it has been cooled to 9°C, place the jar and jacket in another bath maintained at a temperature of minus 15° to minus 18°C. If the material has not ceased to flow when it has been cooled to 6°C, place the jacket and jar in another bath maintained at a temperature of minus 32° to minus 35°C. As soon as the material ceases to flow when the jar is tilted, hold the jar in a horizontal position for exactly 5 seconds. If the sample shows any movement, replace the jar in the jacket and cool the material another 3°C. If the material shows no movement during the 5 seconds, record the reading of the thermometer. Take the pour point as the temperature 3°C above this temperature.

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